Energy, Mines and Resources Canada | Énergie, Mines et Ressources Canada

CANMET

Canada Centre for Mineral and Energy Technology | Centre canadien de la technologie des minéraux et de l'énergie

DIFFERENTIAL THERMAL ANALYSIS OF COAL ASH AND OIL SHALE SAMPLES ON BEHALF OF NEW BRUNSWICK RESEARCH AND PRODUCTIVITY COUNCIL.

W.S. Bowman
Extractive Metallurgy Laboratory

January 1982

This document is an unedited ... report prepared ... for discussion and ... reporting purposes ... represent a final ... the opinion of the ... for Mineral and Energy Technology (CANMET)

... rapport ... rédigé ... pour fin de ... de documentation ... représente nulle... impression définitive de ... du Centre canadien de la technologie des minéraux et de l'énergie (CANMET)

(This report relates essentially to the samples as received)

Project ERP-4.3.0.7.05
Pilot Scale Fluid Bed
Job No. 025131

MICROMEDIA

DECLASSIFIED
DATE 30/07/82
AUTHORIZED BY

CONFIDENTIAL

6 pp

ENERGY RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES
DIVISION REPORT ERP/MSL 82-1 (CF)

DIFFERENTIAL THERMAL ANALYSIS OF COAL ASH AND OIL SHALE SAMPLES ON BEHALF OF NEW BRUNSWICK RESEARCH AND PRODUCTIVITY COUNCIL

by

W.S. BOWMAN*

Samples of coal ash, oil shale and a 70%-30% mixture of the two were received from New Brunswick Research and Productivity Council for differential thermal analysis (DTA) in air to look for evidence of low-melting eutectics. Mixtures of coal and oil shale have shown a tendency to sinter during fluidized-bed combustion, destroying the fluidizing characteristics of the bed.

---

*Technologist, Physical Chemistry Section, Extractive Metallurgy Laboratory, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada.

EXPERIMENTAL

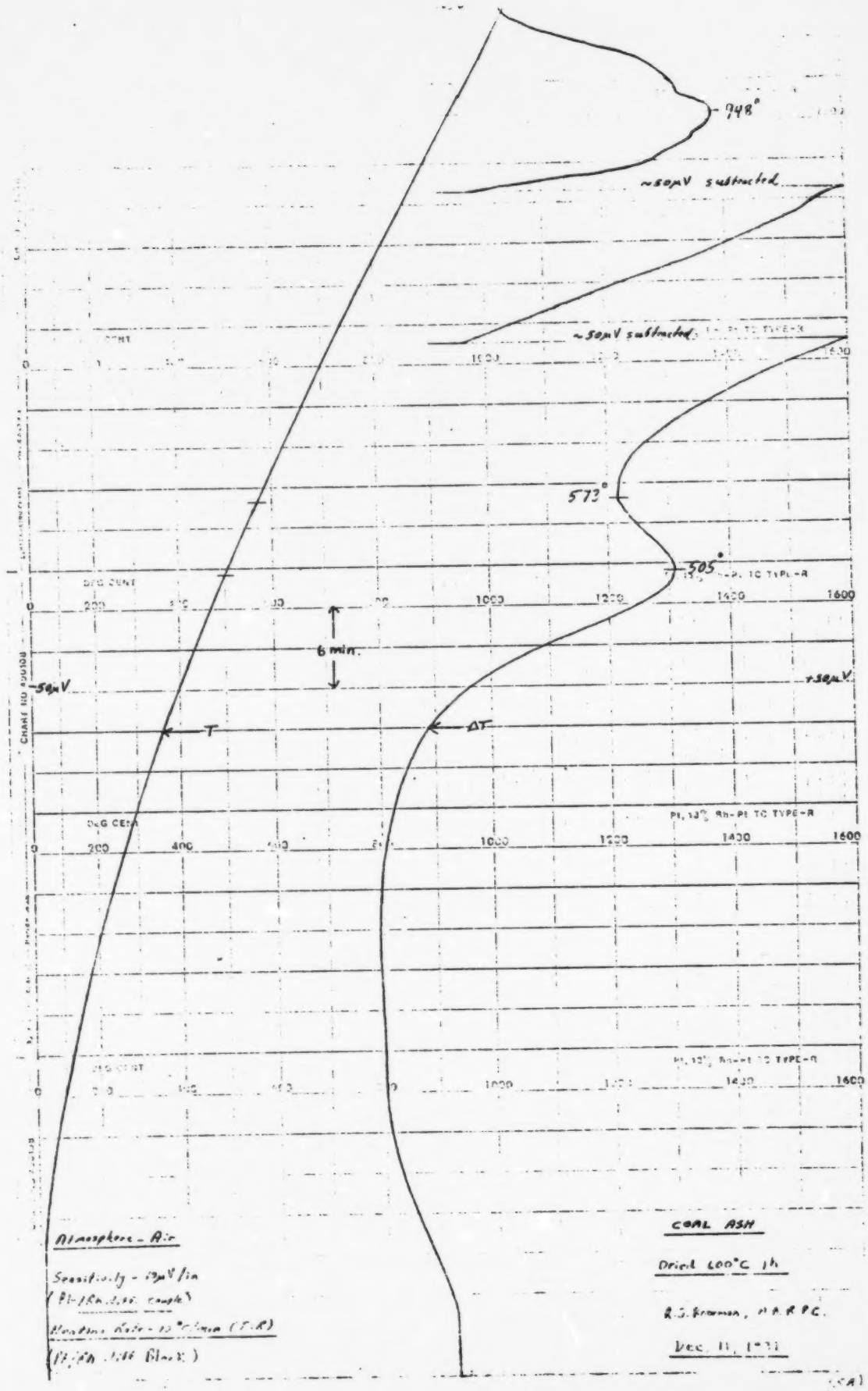
The samples for DTA, weighing about 0.5 g, were lightly packed into a cavity in a Pt/Rh sample block. The cylindrical cavity was 6 mm in diameter and 12 mm deep. An identical cavity, 3 mm from the former, contained the reference material, α-alumina. Pt vs Pt: 13% Rh thermocouples were imbedded in the sample and reference through holes drilled centrally into the wall of each cavity from one end of the holder. The entire arrangement was inserted into mullite protection tube in a Kanthal-wound furnace. The gas inlet and outlet were left open to the atmosphere. A nominal heating rate of 12°C/min was used. The temperature (T) was measured from the reference thermocouple and the difference in temperature between the sample and reference thermocouples (ΔT) was amplified with a Leeds and Northrup 9835-B Microvolt Amplifier. These two signals were recorded on a Leeds and Northrup Speedomax W/L two-pen recorder equipped with a feature that permitted automatic biasing of the latter signal to keep it on scale.

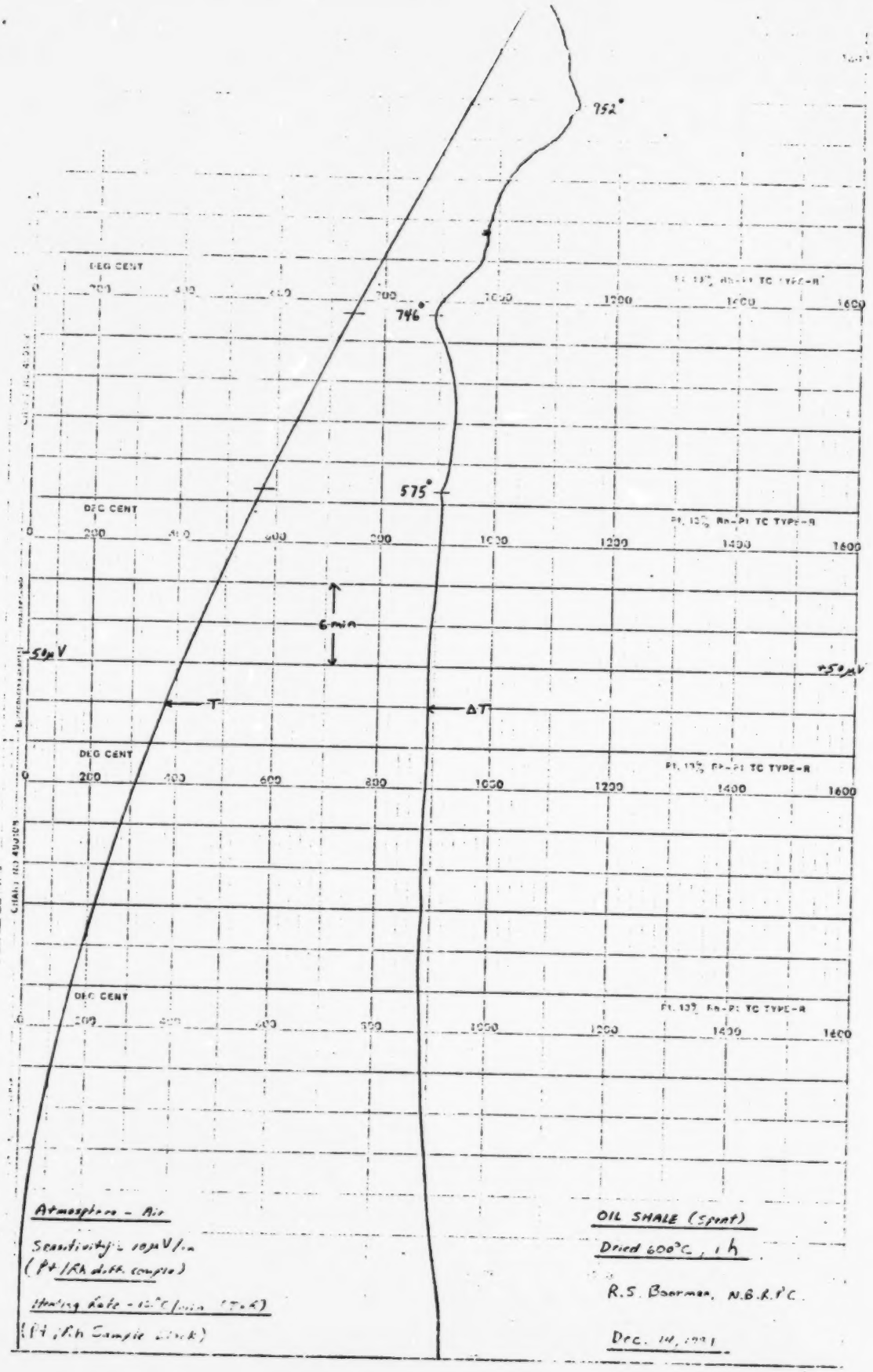
RESULTS

Copies of the DTA charts, reduced to 8½ x 11 inches, are appended to this report. The coal ash sample obviously contained a substantial amount of residual carbon. This began burning at about 400°C resulting in a large two-stage exotherm that persisted to over 1000°C. This effectively masked any other reactions that might have taken place.

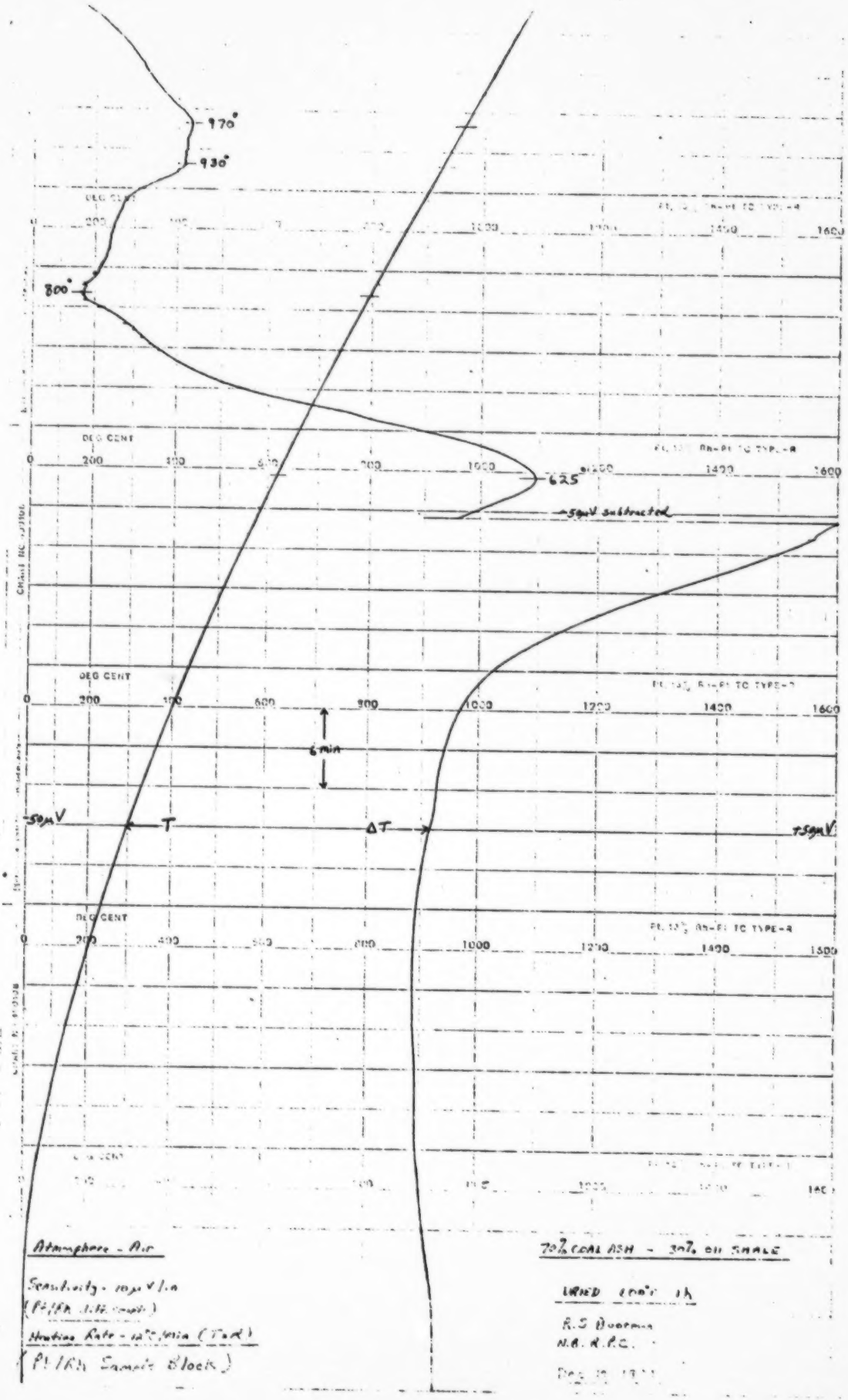
The spent oil shale sample gave relatively small thermal activity. A tiny endotherm at 575° is due to the presence of quartz. The endotherm at 746° could be due to a small eutectic melting. However, the sample after heating to 1050°C, showed no evidence of shrinkage or sintering.

The 70% coal ash-30% oil shale sample gave the same evidence of residual carbon as the pure coal ash sample. It is not possible to see any other reactions below 800°C because of the exotherm due to the burning of carbon. The endothermic peak at 800°C could be due to melting but its magnitude is unknown since it is confounded by the exotherm. The sample, after heating to 1100°C showed no evidence of shrinkage, but did contain a few hard chunks at the bottom of the holder.


948°
~50mV subtracted
~50mV subtracted
573°
505°
6 min.
-50mV
+50mV
T
ΔT
Pt, 13% Rh-Pt TC TYPE-R
Atmosphere - Air
Sensitivity - 5mV/in
(Pt-13Rh-Pt TC type)
Heating Rate - 10°C/min (T.R)
COAL ASH
Dried 100°C 1h
R.J. Brannan, NARPC.
Dec. 11, 1973


752°
746°
575°
DEG CENT
Pt, 13% Rh-Pt TC TYPE-R
6 min
50μV
T
ΔT
Atmosphere - Air
Sensitivity: 10μV/cm
(Pt/Rh diff. couple)
Heating Rate - 12°C/min (T-E)
(Pt/Rh Sample Block)
OIL SHALE (Spent)
Dried 600°C, 1 h
R.S. Boorman, N.B.R.I.C.
Dec. 14, 1971

(3A)


970°
930°
800°
625°
DEG CENT
0
200
600
800
1000
1200
1400
1600
-50μV subtracted
6 min
-50μV
T
ΔT
+50μV
Atmosphere - Air
Sensitivity - 10μV/in
Heating Rate - 12°C/min (T and ΔT)
(Pt/Rh Sample Blocks)
70% COAL ASH - 30% OIL SHALE
LOWER LOOP IN
R.S. Boorman
N.B.R.P.C.